BS EN 61189-5-2:2015

BSI Standards Publication

Test methods for electrical materials, printed boards and other interconnection structures and assemblies

Part 5-2: General test methods for materials and assemblies — Soldering flux for printed board assemblies

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National foreword

This British Standard is the UK implementation of EN 61189-5-2:2015. It is identical to IEC 61189-5-2:2015. It supersedes BS IEC 61189-5-2:2015, which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee EPL/501, Electronic Assembly Technology.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

Published by BSI Standards Limited 2015

ISBN 978 0 580 90018 1

ICS 31.180

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 January 2015.

Amendments/corrigenda issued since publication

Date	Text affected
30 April 2015	This corrigendum renumbers BS IEC 61189-5-2:2015 as BS EN 61189-5-2:2015

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 61189-5-2

March 2015

ICS 31.180

English Version

Test methods for electrical materials, printed boards and other interconnection structures and assemblies - Part 5-2: General test methods for materials and assemblies - Soldering flux for printed board assemblies (IEC 61189-5-2:2015)

Méthodes d'essai pour les matériaux électriques, les cartes imprimées et autres structures d'interconnexion et ensembles - Partie 5-2: Méthodes d'essai générales pour les matériaux et les assemblages - Flux de brasage pour les assemblages de cartes imprimées
(IEC 61189-5-2:2015)

Prüfverfahren für Elektromaterialien, Verbindungsstrukturen und Baugruppen - Teil 5-2: Prüfverfahren für bestückte Leiterplatten - Teil Lötflussmittel
(IEC 61189-5-2:2015)

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CENELEC
European Committee for Electrotechnical Standardization
Comité Européen de Normalisation Electrotechnique
Europäisches Komitee für Elektrotechnische Normung

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

Foreword

The text of document 91/1210/FDIS, future edition 1 of IEC 61189-5-2, prepared by IEC/TC 91 "Electronics assembly technology" was submitted to the IEC-CENELEC parallel vote and approved by CENELEC as EN 61189-5-2:2015.

The following dates are fixed:

• latest date by which the document has to be implemented at national level by publication of an identical national standard or by endorsement	(dop)	2015-11-12
• latest date by which the national standards conflicting with the document have to be withdrawn	(dow)	2018-02-12

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The text of the International Standard IEC 61189-5-2:2015 was approved by CENELEC as a European Standard without any modification.

In the official version, for Bibliography, the following notes have to be added for the standards indicated:

IEC 60068 Series	NOTE	Harmonized as EN 60068 Series.
IEC 60068-1:2013	NOTE	Harmonized as EN 60068-1:2014 (not modified).
IEC 60068-2-20	NOTE	Harmonized as EN 60068-2-20.
IEC 61189-1	NOTE	Harmonized as EN 61189-1.
IEC 61189-2	NOTE	Harmonized as EN 61189-2.
IEC 61189-3	NOTE	Harmonized as EN 61189-3.
IEC 61190-1-2	NOTE	Harmonized as EN 61190-1-2.
IEC 61249-2-7	NOTE	Harmonized as EN 61249-2-7.
IEC 62137:2004	NOTE	Harmonized as EN 62137:2004 (not modified).
ISO 9001	NOTE	Harmonized as EN ISO 9001.

Annex ZA
(normative)

Normative references to international publications with their corresponding European publications

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

NOTE 1 When an International Publication has been modified by common modifications, indicated by (mod), the relevant EN/HD applies.

NOTE 2 Up-to-date information on the latest versions of the European Standards listed in this annex is available here: www.cenelec.eu

Publication	Year	Title	EN/HD	Year
IEC 61189-5	-	Test methods for electrical materials, interconnection structures and assemblies - Part 5: Test methods for printed board assemblies	EN 61189-5	-
IEC 61189-6	-	Test methods for electrical materials, interconnection structures and assemblies - Part 6: Test methods for materials used in manufacturing electronic assemblies	EN 61189-6	-
IEC 61190-1-1	-	Attachment materials for electronic assembly - Part 1-1: Requirements for soldering fluxes for high-quality interconnections in electronics assembly	EN 61190-1-1	-
IEC 61190-1-3	-	Attachment materials for electronic assembly - Part 1-3: Requirements for electronic grade solder alloys and fluxed and non-fluxed solid solders for electronic soldering applications	EN 61190-1-3	-
ISO 9455	Series	Soft soldering fluxes - Test methods	EN ISO 9455	Series
ISO 9455-1	-	Soft soldering fluxes - Test methods - Part 1: Determination of non-volatile matter, gravimetric method	EN 29455-1	-
ISO 9455-2	-	Soft soldering fluxes - Test methods - Part 2: Determination of non-volatile matter, ebulliometric method	EN ISO 9455-2	-

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

TEST METHODS FOR ELECTRICAL MATERIALS, PRINTED BOARDS AND OTHER INTERCONNECTION STRUCTURES AND ASSEMBLIES –

Part 5-2: General test methods for materials and assemblies – Soldering flux for printed board assemblies

FOREWORD

1) The International Electrotechnical Commission (IEC) is a worldwide organization for standardization comprising all national electrotechnical committees (IEC National Committees). The object of IEC is to promote international co-operation on all questions concerning standardization in the electrical and electronic fields. To this end and in addition to other activities, IEC publishes International Standards, Technical Specifications, Technical Reports, Publicly Available Specifications (PAS) and Guides (hereafter referred to as "IEC Publication(s)"). Their preparation is entrusted to technical committees; any IEC National Committee interested in the subject dealt with may participate in this preparatory work. International, governmental and non-governmental organizations liaising with the IEC also participate in this preparation. IEC collaborates closely with the International Organization for Standardization (ISO) in accordance with conditions determined by agreement between the two organizations.

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International Standard IEC 61189-5-2 has been prepared by IEC technical committee 91: Electronics assembly technology.

The text of this standard is based on the following documents:

FDIS	Report on voting
91/1210/FDIS	91/1223/RVD

Full information on the voting for the approval of this standard can be found in the report on voting indicated in the above table.

This publication has been drafted in accordance with the ISO/IEC Directives, Part 2.

This International Standard is used in conjunction with IEC 61189-1:1997, IEC 61189-2:2006, IEC 61189-3:2007.

A list of all parts in the IEC 61189 series, published under the general title *Test methods for electrical materials, printed boards and other interconnection structures and assemblies*, can be found on the IEC website.

The committee has decided that the contents of this publication will remain unchanged until the stability date indicated on the IEC web site under "http://webstore.iec.ch" in the data related to the specific publication. At this date, the publication will be

- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

INTRODUCTION

IEC 61189 relates to test methods for materials or component robustness for printed board assemblies, irrespective of their method of manufacture.

The standard is divided into separate parts, covering information for the designer and the test methodology engineer or technician. Each part has a specific focus; methods are grouped according to their application and numbered sequentially as they are developed and released.

In some instances test methods developed by other TCs (for example, TC 104) have been reproduced from existing IEC standards in order to provide the reader with a comprehensive set of test methods. When this situation occurs, it will be noted on the specific test method; if the test method is reproduced with minor revisions, those paragraphs that are different are identified.

This part of IEC 61189 contains test methods for evaluating robustness of materials or component for printed board assemblies. The methods are self-contained, with sufficient detail and description so as to achieve uniformity and reproducibility in the procedures and test methodologies.

The tests shown in this standard are grouped according to the following principles:

P: preparation/conditioning methods
V: visual test methods
D: dimensional test methods
C: chemical test methods
M: mechanical test methods
E: electrical test methods
N: environmental test methods
X: miscellaneous test methods

To facilitate reference to the tests, to retain consistency of presentation, and to provide for future expansion, each test is identified by a number (assigned sequentially) added to the prefix (group code) letter showing the group to which the test method belongs.

The test method numbers have no significance with respect to an eventual test sequence; that responsibility rests with the relevant specification that calls for the method being performed. The relevant specification, in most instances, also describes pass/fail criteria.

The letter and number combinations are for reference purposes to be used by the relevant specification. Thus "5-2C01" represents the first chemical test method described in IEC 61189-5-2.

In short, in this example, 5-2 is the number of the part of IEC 61189, C is the group of methods, and 01 is the test number.

TEST METHODS FOR ELECTRICAL MATERIALS, PRINTED BOARDS AND OTHER INTERCONNECTION STRUCTURES AND ASSEMBLIES –

Part 5-2: General test methods for materials and assemblies – Soldering flux for printed board assemblies

1 Scope

This part of IEC 61189 is a catalogue of test methods representing methodologies and procedures that can be applied to test printed board assemblies.

This part of IEC 61189 focuses on test methods for soldering flux based on the existing IEC 61189-5 and IEC 61189-6. In addition, it includes test methods of soldering flux for lead free soldering.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

IEC 61189-5, *Test methods for electrical materials, interconnection structures and assemblies – Part 5: Test methods for printed board assemblies*

IEC 61189-6, *Test methods for electrical materials, interconnection structures and assemblies – Part 6: Test methods for materials used in manufacturing electronic assemblies*

IEC 61190-1-1, *Attachment materials for electronic assembly – Part 1-1: Requirements for soldering fluxes for high-quality interconnections in electronics assembly*

IEC 61190-1-3, *Attachment materials for electronic assembly– Part 1-3: Requirements for electronic grade solder alloys and fluxed and non-fluxed solid solders for electronic soldering applications*

ISO 9455 (all parts), *Soft soldering fluxes – Test methods*

ISO 9455-1, *Soft soldering fluxes – Test methods – Part 1: Determination of non-volatile matter, gravimetric method*

ISO 9455-2, *Soft soldering fluxes –Test methods – Part 2: Determination of non-volatile matter, ebulliometric method*

3 Accuracy, precision and resolution

3.1 General

Errors and uncertainties are inherent in all measurement processes. The information given below enables valid estimates of the amount of error and uncertainty to be taken into account.

Test data serve a number of purposes which include

- monitoring of a process;
- enhancing of confidence in quality conformance;
- arbitration between customer and supplier.

In any of these circumstances, it is essential that confidence can be placed upon the test data in terms of

- accuracy: calibration of the test instruments and/or system;
- precision: the repeatability and uncertainty of the measurement;
- resolution: the suitability of the test instrument and/or system.

3.2 Accuracy

The regime by which routine calibration of the test equipment is undertaken shall be clearly stated in the quality documentation of the supplier or agency conducting the test and should meet the requirements of ISO 9001.

The calibration shall be conducted by an agency having accreditation to a national or international measurement standard institute. There should be an uninterrupted chain of calibration to a national or international standard.

Where calibration to a national or international standard is not possible, round-robin techniques may be used and documented to enhance confidence in measurement accuracy.

The calibration interval shall normally be one year. Equipment consistently found to be outside acceptable limits of accuracy shall be subject to shortened calibration intervals. Equipment consistently found to be well within acceptable limits may be subject to relaxed calibration intervals.

A record of the calibration and maintenance history shall be maintained for each instrument. These records should state the uncertainty of the calibration technique (in ± % deviation) in order that uncertainties of measurement can be aggregated and determined.

A procedure shall be implemented to resolve any situation where an instrument is found to be outside calibration limits.

3.3 Precision

The uncertainty budget of any measurement technique is made up of both systematic and random uncertainties. All estimates shall be based upon a single confidence level, the minimum being 95 %.

Systematic uncertainties are usually the predominant contributor and will include all uncertainties not subject to random fluctuation. These include

- calibration uncertainties;
- errors due to the use of an instrument under conditions which differ from those under which it was calibrated;
- errors in the graduation of a scale of an analogue meter (scale shape error).

Random uncertainties result from numerous sources but can be deduced from repeated measurement of a standard item. Therefore, it is not necessary to isolate the individual contributions. These may include

- random fluctuations such as those due to the variation of an influence parameter. Typically, changes in atmospheric conditions reduce the repeatability of a measurement;

- uncertainty in discrimination, such as setting a pointer to a fiducial mark or interpolating between graduations on an analogue scale.

Aggregation of uncertainties: Geometric addition (root-sum-square) of uncertainties may be used in most cases. Interpolation error is normally added separately and may be accepted as being 20 % of the difference between the finest graduations of the scale of the instrument.

$$U_\mathrm{t} = \pm\sqrt{(U_\mathrm{s}^2 + U_\mathrm{r}^2)} + U_\mathrm{i}$$

where

U_t is the total uncertainty;

U_s is the systematic uncertainty;

U_r is the random uncertainty;

U_i is the interpolation error.

Determination of random uncertainties: Random uncertainty can be determined by repeated measurement of a parameter and subsequent statistical manipulation of the measured data. The technique assumes that the data exhibits a normal (Gaussian) distribution.

$$U_\mathrm{r} = \frac{t \times \sigma}{\sqrt{n}}$$

where

U_r is the random uncertainty;

n is the sample size;

t is the percentage point of the t distribution as shown in Table 1;

σ is the standard deviation (σ_{n-1}).

3.4 Resolution

It is paramount that the test equipment used is capable of sufficient resolution. Measurement systems used should be capable of resolving 10 % (or better) of the test limit tolerance.

It is accepted that some technologies will place a physical limitation upon resolution (for example, optical resolution).

3.5 Report

In addition to requirements detailed in the test specification, the report shall detail

a) the test method used;
b) the identity of the sample(s);
c) the test instrumentation;
d) the specified limit(s);
e) an estimate of measurement uncertainty and resultant working limit(s) for the test;
f) the detailed test results;
g) the test date and operators' signature.

3.6 Student's *t* distribution

Table 1 gives values of the factor t for 95 % and 99 % confidence levels, as a function of the number of measurements.

Table 1 – Student's *t* distribution

Sample size	*t* value 95 %	*t* value 99 %		Sample size	*t* value 95 %	*t* value 99 %
2	12,7	63,7		14	2,16	3,01
3	4,3	9,92		15	2,14	2,98
4	3,18	5,84		16	2,13	2,95
5	2,78	4,6		17	2,12	2,92
6	2,57	4,03		18	2,11	2,9
7	2,45	3,71		19	2,1	2,88
8	2,36	3,5		20	2,09	2,86
9	2,31	3,36		21	2,08	2,83
10	2,26	3,25		22	2,075	2,82
11	2,23	3,17		23	2,07	2,81
12	2,2	3,11		24	2,065	2,8
13	2,18	3,05		25	2,06	2,79

3.7 Suggested uncertainty limits

The following target uncertainties are suggested:

a) Voltage < 1 kV: ± 1,5 %
b) Voltage > 1 kV: ± 2,5 %
c) Current < 20 A: ± 1,5 %
d) Current > 20 A: ± 2,5 %

Resistance

e) Earth and continuity: ± 10 %
f) Insulation: ± 10 %
g) Frequency: ± 0,2 %

Time

h) Interval < 60 s: ± 1 s
i) Interval > 60 s: ± 2 %
j) Mass < 10 g: ± 0,5 %
k) Mass 10 g – 100 g: ± 1 %
l) Mass > 100 g: ± 2 %
m) Force: ± 2 %
n) Dimension < 25 mm: ± 0,5 %
o) Dimension > 25 mm: ± 0,1 mm
p) Temperature < 100 °C: ± 1,5 %
q) Temperature > 100 °C: ± 3,5 %
r) Humidity 30 % to 75 % RH: ± 5 % RH

Plating thicknesses

s) Backscatter method: ± 10 %
t) Microsection: ± 2 µm

u) Ionic contamination: ± 10 %

4 C: Chemical test methods

4.1 Test 5-2C01: Corrosion, flux

4.1.1 Object

This test method is designed to determine the corrosive properties of flux residues under extreme environmental conditions. A pellet of solder is melted in contact with the test flux on a sheet metal test piece. The solder is then exposed to prescribed conditions of humidity and the resulting corrosion, if any, is assessed visually.

4.1.2 Test specimen

At least 0,035 g of flux solids, 1 g solder paste, 1 g wire, or 1 g preform with an equivalent amount of solids. Flux solids are defined as the residue from the solid content, flux test described in this 4.1. All solvent shall have been evaporated from the specimen in a chemical fume hood.

4.1.3 Apparatus and reagents

The following apparatus and reagents are needed:

a) solder pot;

b) humidity chamber capable of achieving (40 ± 1) °C and (93 ± 2) % relative humidity;

c) air-circulating drying oven;

d) microscope having minimum 20×;

e) chemicals: All chemicals shall be reagent grade (highly pure, without contamination) and water shall be distilled or deionized: ammonium persulphate; sulphuric acid, % volume (v/v), degreasing agent; acetone, or petroleum ether;

f) analytical balance capable of weighing 0,001 g;

g) copper sheet of a thickness of (0,50 ± 0,05) mm and a purity of 99 %.

4.1.4 Procedures

4.1.4.1 Chemicals

a) Ammonium persulphate (25 % m/v in 0,5 % v/v sulphuric acid). Dissolve 250 g of ammonium persulphate in water and add cautiously 5 ml of sulphuric acid (density 1,84 g/cm^3). Mix, cool, dilute to 1 litre and mix. This solution should be freshly prepared.

b) Sulphuric acid (5 % v/v). To 400 ml of water cautiously add 50 ml of sulphuric acid (density 1,84 g/cm^3). Mix, cool, dilute to 1 l and mix.

4.1.4.2 Test panel preparation

a) Cut a piece of 50 mm × 50 mm from the copper sheet for each test.

b) Form a circular depression in the centre of each test panel 3 mm deep by forcing a steel ball of a diameter of 20 mm into a hole of a diameter of 25 mm to form a cup.

c) Bend one corner of each test panel up to facilitate subsequent handling with tongs.

4.1.4.3 Preconditioning test panels

Immediately before performing the test, precondition as follows using clean tongs for handling.

a) Degrease with a suitable neutral organic solvent such as acetone or petroleum ether.

b) Immerse in 5 % sulphuric acid (by volume) at (65 ± 5) °C for 1 min to remove the tarnish film.

c) Immerse in a solution of 25 % m/v ammonium persulphate (0,5 % v/v sulphuric acid) at (23 ± 2) °C for 1 min to etch the surface uniformly.

d) Wash in running tap water for a maximum of 5 s.

e) Immerse in 5 % sulfuric acid (by volume) at (23 ± 2) °C for 1 min.

f) Wash for 5 s in running tap water, then rinse thoroughly in deionized water.

g) Rinse with acetone.

h) Allow to dry in clean air.

i) Use the test piece as soon as possible or store up to 1 h in a closed container.

4.1.4.4 Preparation of test solder

a) Weigh (1,00 ± 0,05) g specimen of solder for each test and place in the centre of depression of each test panel.

b) Degrease solder specimen with a suitable neutral organic solvent such as acetone or petroleum ether.

c) Solder may be in the form of pellets or by forming tight spirals of solder wire.

4.1.4.5 Test

a) Heat solder pot so that solder bath stabilizes at (235 ± 5) °C in the case of Sn63Pb37 and Sn60Pb40 alloy, or at (255 ± 3) °C for Sn96,5Ag3Cu0,5, or at 35±3 °C higher than the liquidus temperature of any other solder alloy as agreed between the user and the supplier. For solder alloys except Sn63Pb37 and Sn60Pb40, the temperature of the solder pot may be approximately 40 °C higher than the liquid temperature of each alloy.

b) Liquid flux, place 0,035 g of flux solids into the depression in the test panel. Add solder sample.

c) Solder paste, cored wire or cored preform, place 1 g of solder paste, flux-cored wire or cored-preform into the depression in the test panel.

d) Using tongs, lower each test panel onto the surface of the molten solder.

e) Allow the test panel to remain in contact until the solder specimen in the depression of the test panel melts. Maintain this condition for (5 ± 1) s.

f) Carefully examine the test panel at 20× magnification for subsequent comparison after humidity exposure. Record observations, especially any discoloration.

g) Preheat test panel to (40 ± 1) °C for (30 ± 2) min.

h) Preset humidity chamber to (40 ± 1) °C and (93 ± 2) % relative humidity.

i) Suspend each test panel vertically (and separately) in the humidity chamber.

j) Expose panels to the above environment for 72 h (3 days). M (moderately active) and H (highly active) flux may be tested in the cleaned, as well as uncleaned, condition.

4.1.4.6 Evaluation

Carefully examine test panels prior to placing them in the environmental chamber. Note any discoloration.

After the appropriate exposure period, remove test panels from humidity chamber, examine at 20× magnification and compare with observations noted prior to exposure.

Corrosion is described as follows.

- Excrescences at the interfaces of the flux residue and copper boundary or the residues or discontinuities in the residues.
- Discrete white or coloured spots in the flux residues.

An initial change of colour which may develop when the test panel is heated during soldering is disregarded, but subsequent development of green-blue discoloration with observation of pitting of the copper panel is regarded as corrosion.

4.1.5 Additional information

4.1.5.1 Definition of corrosion

For the purposes of this test method, the following is the definition of corrosion: “chemical reaction between the copper, the solder, and the constituents of the flux residues, which occurs after soldering and during exposure to the above environmental conditions."

Colour photos before and after the test are valuable tools in identifying corrosion.

4.1.5.2 Safety

Observe all appropriate precautions on material safety data sheets (MSDS) for chemicals involved in this test method.

4.2 Test 5-2C02: Determination of acid value of liquid soldering flux potentiometric and visual titration methods

4.2.1 Object

This test method specifies two methods for the determination of the acid value of a flux of types L, M or H.

Method A is a potentiometric titration method and is to be considered as the reference method.

Method B is an alternative, visual end-point, titration method.

See ISO 9455 for reference.

4.2.2 Test specimen

A minimum of 2,0 g of liquid flux, 10 g of solder paste, 150 g of cored wire or 10 g of solder preforms.

4.2.3 Apparatus and reagents

4.2.3.1 General

a) Use only reagents of recognized analytical quality and only distilled or deionized water.
b) Ordinary laboratory apparatus.
c) The term “M” represents molarity of a solution and is calculated by taking the moles of solute and dividing by the litres of solution, e.g. 1,00 mole of sucrose (about 342,3 g) mixed into a litre of water equals 1,00 M (1,00 mol/l).

4.2.3.2 Potentiometric titration method (Method A)

a) Tetrabutyl ammonium hydroxide. 0,1 M (0,1 mol/l). Use a commercially available standard solution or one prepared from a commercially available concentrated standard solution by dilution with propan-2-ol. Standardize this solution against an accurately weighed amount of benzoic acid (about 0,5 g) dissolved in dimethylformamide, previously neutralized to thymol blue.
b) Propan-2-ol: neutralized with tetrabutyl ammonium hydroxide solution to a faint pink colour using phenolphthalein as an indicator.
c) Ethanol 96% by volume: neutralized with tetrabutyl ammonium hydroxide solution to a faint pink colour using phenolphthalein as an indicator.

d) Toluene: neutralized with tetrabutyl ammonium hydroxide solution to a faint pink colour using phenolphthalein as an indicator.

e) Ethanol/toluene mixture: mix equal volumes of the ethanol 96 % by volume and toluene.

f) Millivoltmeter or pH meter.

g) Glass electrode.

h) Saturated calomel, or silver chloride/silver, electrode.

i) Magnetic or mechanical stirrer with variable speed drive.

4.2.3.3 Titration with visual end-point (Method B)

a) Ethanol 96 % by volume: neutralized with potassium hydroxide, 0,1 M in alcohol, to a faint pink colour using phenolphthalein as an indicator.

b) Toluene: neutralized with potassium hydroxide, 0,1 M in alcohol, to a faint pink colour using phenolphthalein as an indicator.

c) Ethanol/toluene mixture: mix equal volumes of the ethanol 96 % by volume and toluene.

d) Propan-2-ol: neutralized with potassium hydroxide, 0,1 M in alcohol, to a faint pink colour using phenolphthalein as an indicator.

e) Potassium hydroxide solution: 0,1 M in alcohol. Use a commercially available standard solution or one prepared from a commercially available concentrated standard solution by dilution with ethanol. Standardize this solution against an accurately weighed amount of benzoic acid (about 0,5 g) dissolved in ethanol.

f) Phenolphthalein indicator solution: Add 1 g of phenolphthalein to approximately 50 ml methanol and mix. When dissolved, dilute to 100 ml with methanol and mix.

4.2.4 Procedures

4.2.4.1 Potentiometric titration (Method A)

a) By preliminary experiments, determine whether the specimen is soluble in propan-2-ol, ethanol 96 % by volume, toluene or the ethanol/toluene mixture. If it is not completely soluble in any of these solvents, select the one in which it appears to be the most soluble. If it is equally soluble in all four solvents then use propan-2-ol.

b) Carry out the following procedure, in triplicate, on the flux specimen.

c) Weigh, to the nearest 0,001 g, 2,0 g to 5,0 g of the liquid flux specimen taking steps to prevent loss of volatile matter during the weighing. The larger size specimen is required for very low solids fluxes. Transfer the weighed specimen to a 250 ml low form beaker.

d) Dilute specimen to 100 ml with propan-2-ol, or the selected solvent, according to the solubility characteristics of the flux. Cover with a watch glass and dissolve the flux by gentle agitation.

e) Place the beaker on the stand of the titration assembly with the electrodes, stirrer and burette in position. Adjust the speed of the stirrer to give vigorous stirring without splashing. Titrate with the tetrabutyl ammonium hydroxide solution, adding 1 ml portions and recording the pH, or mV meter readings after each addition. As the end point is approached, reduce the additions of titrant to 0,1 ml and continue titrating past the end point.

f) Plot the pH, or potential values against the volume of titrant added to obtain the titration curve. The point of inflection of the curve corresponds to the end-point of the titration.

g) Carry out a blank determination, using all reagents, for comparison purposes.

4.2.4.2 Visual titration (Method B)

a) By preliminary experiments, determine whether the specimen is soluble in propan-2-ol, ethanol 96 % by volume, toluene or the ethanol/toluene mixture. If it is not completely soluble in any of these solvents, select the one in which it appears to be the most soluble. If it is equally soluble in all four solvents then use ethanol as the selected solvent.

b) Carry out the following procedure, in triplicate, on the flux specimen.

c) Weigh, to the nearest 0,001 g sufficient of the flux specimen to correspond to approximately 1 g of non-volatile matter in accordance with test method 6C03, taking steps in the case of liquid flux specimens to prevent loss of volatile matter during the weighing.

d) Transfer the weighed specimen to a suitable flask or beaker and add 100 ml of the selected solvent. Stir until the specimen has dissolved as completely as possible. Do not heat.

e) Add 3 drops of phenolphthalein indicator and titrate with the potassium hydroxide (4.2.3.3) until a faint pink colour persists throughout the titrated solution for 15 s.

f) Carry out blank determination, using all reagents, for comparison purposes.

4.2.4.3 Calculation of results

a) The acid value is expressed in milligrams of potassium hydroxide per gram of non-volatile matter, regardless of the alkali used to perform the titration.

b) The acid value (expressed in milligrams of potassium hydroxide per gram of non-volatile matter) is given by:

$$\frac{56{,}11\,VM}{mS}$$

where

V is the volume, in ml, of alkali used (tetrabutyl ammonium hydroxide for method A, potassium hydroxide for method B);

M is the molarity of the alkali used;

m is the mass, in grams of the specimen taken;

S is the percentage non-volatile matter determined as described in test method 6C03 of this standard.

The acid value (expressed in milligrams of potassium hydroxide per gram of flux) is given by:

$$\frac{56{,}11\,VM}{m}$$

The acid value of the flux under test is calculated as the mean of the results obtained on each of the three test specimens.

4.2.5 Additional information

Safety: operator should be trained and familiar with the hazards inherent to the chemicals being used and analysed. Proper personal safety equipment, such as safety glasses, gloves and splash apron, as well as adequate ventilation, should be used.

4.3 Test 5-2C03: Acid number of rosin

Under consideration.

4.4 Test 5-2C04: Determination of halides in fluxes, silver chromate method

4.4.1 Object

This test method is designed to determine the presence of chlorides and bromides in soldering flux by visual examination after placement of the flux on test paper.

4.4.2 Test specimen

The test specimen shall consist of a minimum of 100 ml of liquid flux, a representative container of solder paste, paste flux for reflow soldering, extracted solder preform flux or extracted flux-cored wire.

4.4.3 Apparatus and reagents

a) Six pieces of silver chromate test paper 51 mm × 51 mm.

b) 0,25 l of reagent grade (highly pure, without contamination) propan-2-ol.

4.4.4 Procedure

4.4.4.1 Preparation

a) The silver chromate paper is extremely light sensitive and shall be stored in a closed container away from light until used for testing.

b) To avoid contamination, the paper shall be handled with forceps and shall never be touched with bare hands.

4.4.4.2 Test for liquid flux or flux extract solution

a) Place one drop of test flux or flux extract (approximately 0,05 ml) on each piece of silver chromate test paper. Allow the droplet to remain on each test paper for a minimum of 15 s.

b) After the 15 s, immediately immerse each test paper in clean propan-2-ol to remove the residual organic materials.

c) Allow each test paper to dry for 10 min, then examine for colour change.

4.4.4.3 Test for paste flux or solder paste flux as obtained from the supplier

a) Clean a glass microscope slide with propan-2-ol and air dry.

b) Moisten a piece of silver chromate reagent paper of suitable size with deionized water.

c) Apply the wet paper to the glass slide and remove the excess water with blotting paper.

d) Using a spatula, apply a thin coating of the paste flux or solder paste flux directly to the moist reagent paper.

e) Allow the paste flux or solder paste flux to remain in contact with the paper for 1 min, then remove the flux with propan-2-ol without disturbing the paper.

4.4.5 Evaluation

Carefully examine each test sheet for possible colour change. A change to off-white or yellow-white indicates the presence of chlorides or bromides (see Figure 1).

Interferences:

a) A number of chemicals may cause test failures. (Representative examples are, but are not limited to, amines, cyanides, isocyanates, etc.)

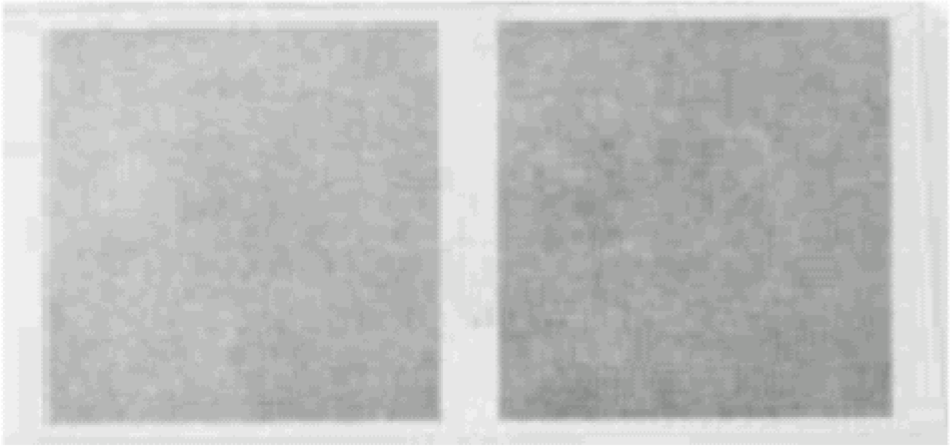
b) Certain acidic solutions may react with the reagent paper to provide a colour change similar to that obtained with chlorides and bromides. When a colour change is observed, it is advisable to check the acidity of the affected area by means of a pH indicating paper. If pH values of less than 3 are obtained, the presence of chlorides and bromides should be verified by other analytical means.

4.4.6 Additional information

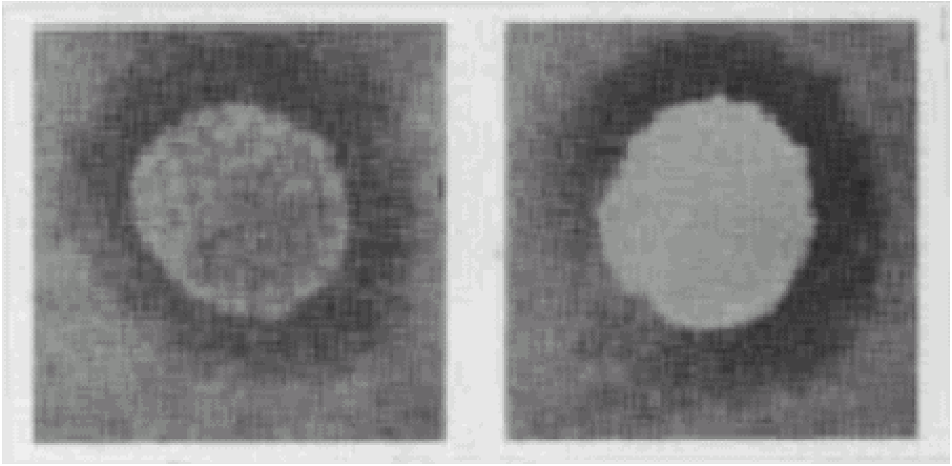
Safety: Observe all appropriate precautions on the material safety data sheets (MSDS) for chemicals involved in this test method.

Source for silver chromate test paper:

Quantek, PO Box 136, Lyndhurst, NJ 07071, (201) 935-4103



PASS



FAIL

Figure 1 – Chlorides and/or bromides test results

4.5 Test 5-2C05: Solids content, flux

4.5.1 Object

This test method is designed to determine the residual solids content of the liquid flux after evaporation of the volatile chemicals from the liquid flux; typically 15 % by weight minimum.

4.5.2 Test specimen

The test specimen shall consist of a minimum of 6 g per test of liquid flux or flux extracted from solder paste, solder preforms or flux-cored wire.

4.5.3 Apparatus and reagents

a) a circulating air drying oven capable of maintaining a temperature of (110 ± 5) °C;

b) analytical balance capable of weighing 0,000 1 g;

c) glass pipettes;

d) glass petri dishes, 30 ml capacity;

e) silica gel desiccant, or equivalent, in a glass desiccator.

4.5.4 Procedures

4.5.4.1 Instruction

Carry out the following procedures in triplicate.

4.5.4.2 Preparation

a) Dry three empty glass petri dishes in the drying oven, then cool in the desiccator to room temperature.

b) Weigh each dish to the nearest 0,001 g.

4.5.4.3 Test

a) Pipette approximately 6 g of test flux specimen into each petri dish and weigh to the nearest 0,001 g.

b) Heat in the drying oven for 1 h, then re-weigh after allowing the specimen to come to room temperature.

c) Repeat heating and drying procedure until the weight is constant to within 0,005 g.

4.5.5 Evaluation

Calculate the residual solids as follows:

$$C_\mathrm{s} = \frac{100 \times m_2}{m_1}$$

where

C_s are the residual solids;

m_2 is the mass of residual after drying, in g;

m_1 is the mass of original test flux specimen, in g.

4.5.6 Additional information

4.5.6.1 Specimen size

Larger specimen sizes may be required to obtain accurate data on low solids (<15 %) fluxes.

4.5.6.2 Safety

Observe all appropriate precautions on the material safety data sheets (MSDS) for chemicals involved in this test method.

4.6 Test 5-2C06: Quantitative determination of halide content in fluxes (chloride and bromide)

4.6.1 Object

This test method is designed to determine the halide content of fluxes attributable to chlorides and bromides. The halide content is reported as the weight percentage of chloride to the solid (non-volatile) portion of the flux or as milliequivalent per gram of flux solids. A specimen of flux or flux extract is titrated to an end-point and the percentage chloride or meq/g of halides is calculated.

Method A is an alternative, visual end- point, titration method.

Method B is a potentiometric titration method.

4.6.2 Test specimen

4.6.2.1 Visual titration (Method A)

A minimum of 100 ml of liquid flux, 10 ml to 50 ml flux extract of known solids content from solder paste, solder preforms or flux-cored wire.

4.6.2.2 Potentiometric titration method (Method B)

A minimum of 200 ml of liquid flux, containing (5,0 ± 0,1) g flux extracted of known solids content from solder paste, solder preforms or flux-cored wire.

4.6.3 Apparatus and reagents

4.6.3.1 General

a) Use only reagents of recognized analytical quality and only distilled or deionized water.

b) Ordinary laboratory apparatus.

c) The term “M” represents molarity of a solution and is calculated by taking the moles of solute and dividing by the litres of solution, e.g. 1,00 mole of sucrose (about 342,3 g) mixed into a litre of water equals 1,00 M (1,00 mol/l).

   A normal solution (N) contains 1 g of solute per litre of solution.

   A molar solution (M) contains 1 mole of solute per litre of solution.

   Examples:

   A 0,2 M solution of NaCl contains 0,2 moles of NaCl per litre.

   A 3 N solution of NaCl contains 3 moles of NaCl per litre.

4.6.3.2 Visual titration (Method A)

a) hot plate with magnetic stirrer;

b) analytical balance capable of reading to 0,001 g;

c) pipettes;

d) burettes;

e) 100 ml beakers, pyrex®[1];

f) 125 ml separatory funnel;

g) 125 ml Erlenmeyer flasks;

h) 1 000 ml volumetric flasks;

i) 0,1 N silver nitrate, standardized: dissolve 17,000 g silver nitrate in deionized water and dilute to 1 000 ml in a volumetric flask;

j) 1 M (1 mol/l) sodium hydroxide: 40,0 g of sodium hydroxide diluted to 1 000 ml with deionized water in a volumetric flask;

k) 0,2 M (0,2 mol/l) nitric acid: add 12,6 ml concentrated (16 M) nitric acid to deionized water and dilute to 1 000 ml in a volumetric flask;

l) 1 M (1 mol/l) potassium chromate:194 g diluted to 1 000 ml using deionized water in a volumetric flask;

m) 0,03 M (0,03 mol/l) phenolphthalein solution (reagent grade);

n) chloroform (reagent grade);

o) deionized water.

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[1] Pyrex is the trade name of a product supplied by Corning Incorporate or licensees. This information is given for the convenience of users of this standard and does not constitute an endorsement by IEC of the product named. Equivalent products may be used if they can be shown to lead to the same results.

4.6.3.3 Potentiometric titration (Method B)

a) millivolt meter;

b) electrode potassium hydroxide – platinum, platinum-platinum, or silver nitrate-silver electrodes;

c) magnetic stirrer – number of revolutions adjustable;

d) dryer, adjustable to a temperature of (100 ± 5) °C and able to maintain this temperature within the tolerance limits;

e) balance with sensitivity of 0,000 1 g;

f) general purpose experimental device;

g) general purpose goods – only analysis reagent and deionized water;

h) propan-2-ol (reagent grade);

i) standard silver nitrate solution N/20 (0,05 M) available on market or a solution made as follows: dissolve 8,494 g silver nitrate highly pure in deionized water and dilute to 1 000 ml in a volumetric flask.

4.6.4 Procedure

4.6.4.1 Visual titration (Method A)

4.6.4.1.1 Rosin/resin fluxes specimen preparation

a) In a tared 100 ml beaker, accurately weigh about 3 g to 5 g of flux specimen on an analytical balance.

b) Quantitatively transfer the flux specimen to a 125 ml separatory funnel using three 10 ml aliquots of chloroform.

c) Add 15 ml of deionized water to the funnel and shake the funnel for 10 s.

d) Allow the funnel to stand until the layers completely separate.

e) Draw off the bottom (chloroform) layer into a beaker and save for the next extraction.

f) Transfer the top (water) layer to a 125 ml Erlenmeyer flask.

g) Transfer the chloroform layer from the beaker to the funnel and repeat the extraction with 15 ml of water two more times, each time adding the water extract portion to the flask.

h) Heat the water extract in the Erlenmeyer flask using a steam bath to expel any chloroform which may be present.

i) Do not heat above 80 °C. Allow for solution to cool to room temperature.

4.6.4.1.2 Organic and inorganic flux specimen preparation

a) In a tared 125 ml Erlenmeyer flask, accurately weigh about 3 g to 5 g of flux specimen on analytical balance.

b) Add 50 ml of deionized water.

c) Add two drops of 0,03 M phenolphthalein solution to the Erlenmeyer flask.

d) Add 1 M sodium hydroxide until the solution turns red. Add 0,2 M nitric acid dropwise until the red colour is just completely discharged.

e) Dilute to about 60 ml with deionized water.

f) Add six drops of 1 M potassium chromate and titrate with standardized 0,1 N silver nitrate to the red-brown end point.

4.6.4.2 Potentiometric titration method (Method B)

4.6.4.2.1 Resin flux cored solder procedure of test

a) Use the dried product itself as test piece after washing the surface with acetone and rinsing first with deionized water and then with propan-2-ol.

b) Measure and cut off solder to produce (5,0 ± 0,1) g of flux, and cut it into chips of 2 mm to 3 mm length.

c) Measure the mass to within an accuracy of 0,001 g and put those chips into a beaker of 300 ml and add 50 ml propan-2-ol.

d) Shake the beaker for about 15 min at normal temperature with a watch dish on, for extracting flux. When flux has dissolved completely, pour the supernatant gently into a 300 ml beaker.

e) Wash the chipped solders with 30 ml propan-2-ol two to three times. Then add this washed solution to the extracted solution, making the total volume equal to 200 ml as test specimen.

f) The chipped solders shall be dried for 1 h in a dryer at (100 ± 5) °C.

g) After cooling, measure the mass to within an accuracy of 0,001 g. The difference between the masses of first measured and chipped solder after extraction of flux shall be the mass of flux.

h) Putting an electrode into the beaker, place the beaker on a magnetic stirrer. Stir strongly preventing spattering, and titrate with silver nitrate standard solution.

i) Measure the potential at every 1 ml, read the potential at every 0,1 ml toward the end of this titration. The point at which the potential changes sharp shall be the end point. For comparison purpose, the blank test shall be carried out for the whole process.

4.6.4.2.2 Solder paste, liquid flux and solid flux

a) Measure a mass of (5,0 ± 0,1) g of flux to within an accuracy of 0,001 g and put it into a 300 ml beaker.

b) Add 200 ml propan-2-ol and stir it at ambient temperature, extracting as much flux as possible. This solution is the test specimen.

c) Put an electrode into the beaker, place the beaker on a magnetic stirrer. Stir strongly preventing spattering, and titrate with silver nitrate standard solution.

d) Measure the potential at every 1 ml, read the potential at every 0,1 ml towards the end of this titration. The point at which the potential changes sharp shall be the end point. For comparison purposes, the blank test shall be carried out for the whole process.

This mass of specimen should be applied to the solders containing halide of more than 0,1 % to 1,0 %. As for the solders containing halide other than above, the figures shown in Table 2 should be applied.

Table 2 – Relation between halide content and mass of specimen

Content of halide mass %	0,05 or less	Over 0,05 0,1 or less	Over 0,1 1,0 or less	Over 1,0
Mass of specimen (g)	50	20	5	1

4.6.5 Calculations

4.6.5.1 Visual titration (Method A)

Calculate the percentage of halides as chloride based on flux solids content, using the following formula:

$$\text{Halides, as \% chlorides} = \frac{3{,}55\, VN \times 100}{mS}$$

Calculate halides milliequivalent per gram of flux solids (non-volatiles) using the following formula:

$$\text{Halides, meq/g solids} = \frac{V \times N \times 100}{mS}$$

where

V is the volume of 0,1 N silver nitrate in ml;

N is the normality of silver nitrate solution;

m is the mass of flux specimen in g;

S is the percentage of solids (non-volatiles) of the flux.

4.6.5.2 Potentiometric titration method (Method B)

The halide content shall be expressed by the content of chlorine (mass %) and shall be calculated by the following formula:

$$H = \frac{3{,}55 \times Mn \times fn \times Vn}{m}$$

where

H is the chlorine content in flux (mass %);

Vn is the volume of silver nitrate (ml);

Mn is the concentration of silver nitrate (mol/l);

fn is the power value of silver nitrate (see note);

m is the mass of specimen (g).

The halide content shall be expressed by the mean value of chlorine content in the flux measured on three specimens.

Power value is determined by the following method. Measure 2,922 g of sodium chloride (reagent grade) to within an accuracy of 0,001 g. Put it into a 1 000 ml flask and dilute with deionized water filled up to the scale line. Exactly 20 ml of this solution is then poured into a 300 ml beaker and titrated with silver nitrate based on the same procedure mentioned above. The power value of silver nitrate solution can be calculated from the formula:

$$fn = \frac{20{,}0 \times m}{y \times 2{,}922}$$

where

m is the mass of special class sodium chlorine (g);

y is the volume of titrated silver nitrate (M).

4.6.6 Report

The halide content is reported as the weight percentage of chloride to the solid (non-volatile) portion of the flux or as milliequivalent per gram of flux solids. A specimen of flux or flux extract is tritrated to an end-point and the percentage chloride or milligram of halides is calculated.

4.6.7 Additional information

Safety: Observe all appropriate precautions on the material safety data sheets (MSDS) for chemicals involved in this test method.

4.7 Test 5-2C07: Qualitative analysis of fluorides and fluxes by spot test

4.7.1 Object

This test method is designed to determine the presence of fluoride(s) in soldering flux by visual examination after placement of a drop of liquid test flux in a zirconium-alizarin purple solution.

4.7.2 Test specimen

a) A minimum of 10 ml of a specimen from a production of liquid flux, solder paste flux, paste flux or extracted flux from preform or wire, furnished in a new or clean glass container.

b) For paste flux or solder paste flux, dilute the specimen in propan-2-ol or another suitable solvent. The solvent used in dilution/extraction should be water miscible.

4.7.3 Apparatus and reagents

4.7.3.1 Apparatus

a) White spot plate.

b) Glass droppers.

c) Glass rods.

4.7.3.2 Reagents

a) Zirconium nitrate solution: 0,05 g dissolved in 50 ml of deionized water;

b) Sodium alizarin sulfate solution: 0,05 g dissolved in 50 ml of deionized water.

4.7.4 Procedure

4.7.4.1 Preparation

Prepare a fresh zirconium-alizarin purple solution in three spots of the white spot plate by adding one drop each of zirconium nitrate and sodium alizarin sulfate solutions.

4.7.4.2 Test

a) Add one drop of the test flux to each of the spots.

b) Mix each spot with a clean glass rod.

c) Examine for any colour change.

4.7.4.3 Evaluation

A change in colour of the solution from purple to yellow is an indication of the presence of fluoride(s).

4.7.4.4 Additional information

Safety: Observe all appropriate precautions on the material safety data sheets (MSDS) for chemicals involved in this test method.

4.8 Test 5-2C08: Quantitative determination of fluoride concentration in fluxes

4.8.1 Object

This test method is used to determine the concentration of fluorides in soldering flux. The fluoride content is reported as the weight percentage of fluoride to the non-volatile portion of the flux. Ion specific electrodes are used for the determination.

4.8.2 Test specimen

a) A minimum of 100 ml of a specimen from a production of liquid rosin flux, or prepared flux solution furnished in a new or clean glass container.

b) For paste flux and solder paste flux, 100 ml of the diluted material.

c) For preform and cored wire, 100 ml of the extracted material.

4.8.3 Apparatus and reagents

4.8.3.1 Apparatus

a) Autocalibrating ion analyser.

b) Fluoride specific electrode.

c) Double junction reference electrode.

d) Magnetic stirrer and stir bars.

e) 250 ml and 500 ml beakers, glass or polypropylene.

f) Funnel stand and funnel.

g) 50 ml graduated cylinders.

h) 100 ml and 1 000 ml volumetric flasks.

i) 10 ml and 100 ml pipettes.

j) Analytical balance capable of weighing 0,001 g.

4.8.3.2 Reagents

a) Deionized water.

b) Sodium fluoride, reagent grade (highly pure, without contamination).

c) Chloroform, reagent grade.

d) Fluoride analysis solution.

e) Ashless filter paper.

4.8.4 Procedure

4.8.4.1 Specimen preparation: water soluble flux

Depending on the expected concentration of fluoride, use the following amount of specimen and deionized water as shown in Table 3.

Table 3 – Mixing ratio from specimen size to water quantity

Fluoride ppm	**Specimen** ml	**Deionized water** ml
0,1 – 0,0	25	50
1,0 – 10,0	5	50

a) Take the beaker and add the specimen; record the weight of the flux to 0,001 g.

b) Add deionized water and mix with stirrer for 10 min; stop mixing and allow to stand for 5 min.

c) Pour solution through filter paper in funnel into a clean beaker. Allow specimen to drain completely.

4.8.4.2 Specimen preparation: non-water soluble flux

Depending on the expected concentration of fluoride, use the following amount of specimen and chloroform as shown in Table 4.

Table 4 – Specimen size to chloroform mixture

Fluoride ppm	**Specimen** ml	**Chloroform** ml
0,1 – 1,0	25	25
1,0 – 10,0	5	5

a) Take the beaker and add the specimen; record the weight of the flux to 0,001 g.

b) Add chloroform and 50 ml deionized water and mix with stirrer for 10 min; stop mixing and allow to stand for 5 min.

c) Carefully decant the water solution through filter paper in funnel into a clean beaker. Allow specimen to drain completely.

4.8.4.3 Standard solutions

a) 10^{-6} fluoride (0,001 %): This standard can be purchased ready-to-use or a solution can be prepared. Weight 0,022 1 g (dried to 140 °C) reagent grade NaF. Add 500 ml deionized water. Dilute to 1 l in a volumetric flask.

b) $1,0^{-6}$ fluoride (0,000 1 %): This standard can be purchased ready-to-use or a solution can be prepared. Weight 0,002 3 g (dried to 140 °C) reagent grade NaF. Add 500 ml deionized water. Dilute to 1 l in a volumetric flask. Another method is to dilute 100 ml, 10^{-6} fluoride standard with 450 ml deionized water and dilute to 1 l in a volumetric flask.

c) $0,1^{-6}$ fluoride (0,000 01 %). This standard is prepared by diluting 100 ml, $1,0^{-6}$ fluoride standard with 450 ml deionized water and dilute to 1 l in a volumetric flask. Another method is to dilute 10 ml, 10^{-6} fluoride standard with 495 ml deionized water and dilute to 1 l in a volumetric flask.

4.8.4.4 Double reference electrode filling solutions

a) Inner chamber filling solution (green): Solution saturated with AgCl.

b) Outer chamber filling solution: 10 % KNO3.

4.8.4.5 Test

a) For accurate results, allow all specimens and standards to reach the same temperature before attempting a measurement. Stir the specimens and standards while a measurement is being made.

b) Prepare two standards that differ in concentration by a factor of 10. The standards should bracket the expected specimen concentration. Select concentration mode and STD 1 function. Significant digit selection is accomplished by pressing the X10 key the required number of times.

c) Dilute 50 ml of the two standard solutions, each with 50 ml of deionized water.

d) While stirring with a magnetic stirrer, immerse electrodes in the more dilute standard solution. Adjust the display using arrows or X10 keys to the correct value. When "Ready" illuminates, press "Enter." The display automatically proceeds to "STD 2."

e) Remove and rinse electrodes with deionized water. Immerse electrodes in the more concentrated standard solution. Adjust the display to read the correct value for STD 2. When "Ready" illuminates, press "Enter." The display automatically proceeds to "Sample" function.

f) To the specimen prepared per instructions for water soluble flux, add 25 ml total ionic strength adjustment buffer. While stirring, immerse electrodes. When display indicates "Ready," record indicated value.

4.8.4.6 Evaluation

The indicated value is equal to the parts per million ($^{-6}$) fluoride. To obtain weight percentage of fluoride as percentage of the non-volatile content, use the following formula:

$$\text{Fluoride \% of solids} = \frac{I}{(mS)\,100}$$

where

I is indicated value in parts per million;

m is the mass of the flux specimen in g;

S is the percentage of solids (non-volatile chemicals) of the flux.

4.8.5 Additional information

4.8.5.1 Calibration and limits

a) Re-calibration should be carried out every few hours to compensate for electrode drift.

b) Reproducibility is typically ±2 % obtainable with hourly recalibration. Temperature fluctuation, drift and noise will limit reproducibility.

c) Detection limits are 0,02 ppm minimum and no known maximum. A list of possible interfering ions has not yet been determined for this electrode method.

4.8.5.2 Safety

The operator should be trained and familiar with the hazards inherent to the chemicals being used and analysed. Proper personal safety equipment such as safety glasses, gloves and splash apron as well as adequate ventilation should be used.

Using the same batch of deionized water for dilutions will eliminate the need to run blanks.

4.8.6 Informative references

Autocalibrating ion analyser – Orion EA 920

Fluoride specific electrode – Orion 94-09-00

Double junction reference electrode – Orion 90-02

Whatman #41 ashless filter paper

10^{-6} fluoride solution – Orion, catalogue #040908

$1,0^{-6}$ fluoride solution – Orion, catalogue #040906

TISAB (total ionic strength adjustment buffer) – Orion catalogue #940909

AgCl solution – Orion catalogue #900002

10 % KNO3 solution – Orion catalogue #900003

4.9 Test 5-2C09: Specific gravity

4.9.1 Object

This test specifies the method for measuring the specific gravity of liquid flux.

4.9.2 Test specimen

The test specimen is a sample of the liquid flux product itself.

4.9.3 Apparatus

a) Tall glass beaker (e.g. a measuring cylinder approximately 200 ml) for liquid flux.

b) Hydrometer.

c) Glass thermometer.

d) Controlled temperature water bath adjustable to (20 ± 1) °C.

4.9.4 Test procedure

a) Clean and dry a circular tall glass beaker for measurement.

b) Measure approximately 150 ml of the specimen and place in the beaker.

c) Place the beaker in the water bath at (20 ± 1) °C.

d) Place the hydrometer and thermometer into the beaker.

e) Read the scale at the upper edge of the meniscus in the hydrometer when the temperature of specimen stabilizes at 20 °C (see Figure 2).

4.9.5 Evaluation

Determine the density from a hydrometer reading.

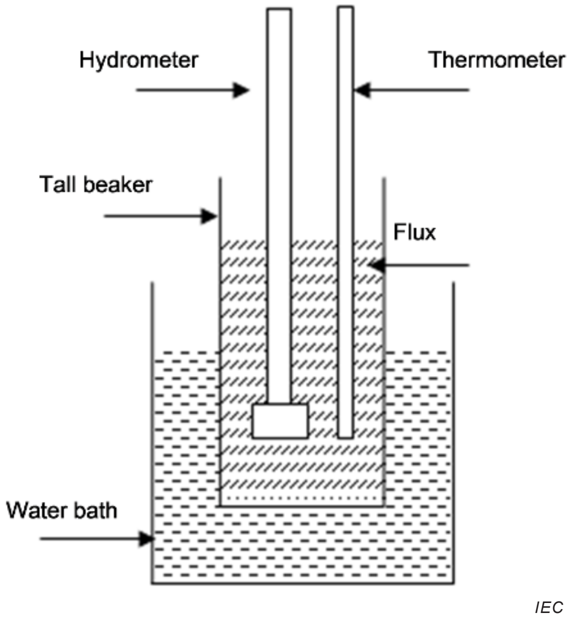


Figure 2 – Test equipment of specific gravity (hydrometer reading)

4.10 Test 5-2C10: Flux induced corrosion (copper mirror method)

4.10.1 Object

This test method is designed to determine the removal effect the flux has (if any) on the bright copper mirror film which has been vacuum deposited on clear glass.

4.10.2 Test specimen

A minimum of 100 ml of liquid flux, a representative container of solder paste, reflowed solder paste flux, extracted solder preform flux or extracted fluxcored wire.

4.10.3 Apparatus and reagents

a) 0,5 l of control standard rosin flux, class A, type II, grade WW, of LLL-R-626.

b) 0,5 l of reagent grade (99 % pure) propan-2-ol.

c) A vacuum deposition system or the means to procure glass test panels having a copper mirror coating as described in point c) of 4.10.4.1 below.

d) 0,5 l of reagent grade 0,5 % solution of ethylene diamine tetra acetic acid (EDTA).

e) 0,5 l of reagent grade ethanol or methanol.

f) 100 ml medicine bottle with dropper.

g) Test cabinet capable of achieving (23 ± 2) °C and (50 ± 5) % relative humidity.

h) Glass slides.

i) A relative humidity gauge with an accuracy of ±2 % or better, to continuously monitor the test environment. The gauge should be calibrated periodically.

4.10.4 Procedure

4.10.4.1 Preparation

a) Preparation of control standard flux: dissolve 35 g rosin into 100 ml of reagent grade 99 % propan-2-ol and stir thoroughly.

b) Preparation of temperature/humidity chamber: when acid or salt solutions are used, the environment shall be monitored for a minimum of 48 h prior to exposing the copper mirror test panels, to assure compliance with the (50 ± 5) % relative humidity requirement.

c) Preparation of copper mirror test panels: apply by vacuum deposition, a film of copper metal on one surface of a flat glass sheet or clear, polished glass. Apply a uniform thickness of approximately 50 nm and assure that the finished mirror permits (10 ± 5) % transmission of normal incident light of nominal wave length of 500 nm. This may be determined using a suitable photoelectric spectrophotometer. Commercially available copper mirrors meeting the above specifications are acceptable.

   Prevent oxidation of the copper mirror by storing in a closed container which has been flushed with nitrogen. Immediately before testing, immerse the copper mirror in a 5 g/l solution of EDTA for copper oxide removal. Mirrors stored in a non-oxidizing environment, do not require cleaning with the EDTA solution prior to testing. The cleaning step shall be used if test results are in dispute. Rinse thoroughly in running water, immerse in clean ethanol or methanol and dry with clean, oil free air. Carefully examine the mirror before testing. There shall be no oxide.

4.10.4.2 Test

a) Place the copper mirror test panel on a flat surface, mirror side up, and protect from dust and dirt at all times. Place one drop of test flux or extract to be tested (approximately 0,05 ml) on each copper mirror test panel. Do not allow the dropper to touch the test panel. Solder paste shall be applied directly to the copper mirror without scratching it; the amount of solder paste shall be approximately 0,5 mm in thickness and 8 mm in diameter. (It has been determined that significant variations from this quantity have little effect for most materials).

b) At the same time, place one drop of the control standard flux adjacent to the test flux. Do not allow drops to touch.

c) Place test panels in a horizontal position in the dust-free cabinet at (23 ± 2) °C and (50 ± 5) % relative humidity for (24 ± 0,5) h.

d) At the end of the 24 h period, remove the test panels and remove the test flux and control standard fluxes by immersion in clean propan-2-ol.

4.10.5 Evaluation

a) Carefully examine each test panel for possible copper removal or discoloration.

b) If there is any complete removal of the copper film as evidenced by the background showing through the glass, the test flux has failed the L category. Complete removal of the copper only around the perimeter of the drop defines the flux as M. Complete removal of the copper places the flux in the H category (see Figure 3).

c) If the control flux fails, repeat the entire test using new copper mirror test panels.

d) Discoloration of the copper film due to a superficial reaction or only a partial reduction of the copper film thickness is not considered as failure.
e) A number of chemicals can cause failure of copper mirror: free halides, stronger organic and inorganic acids and free amines.

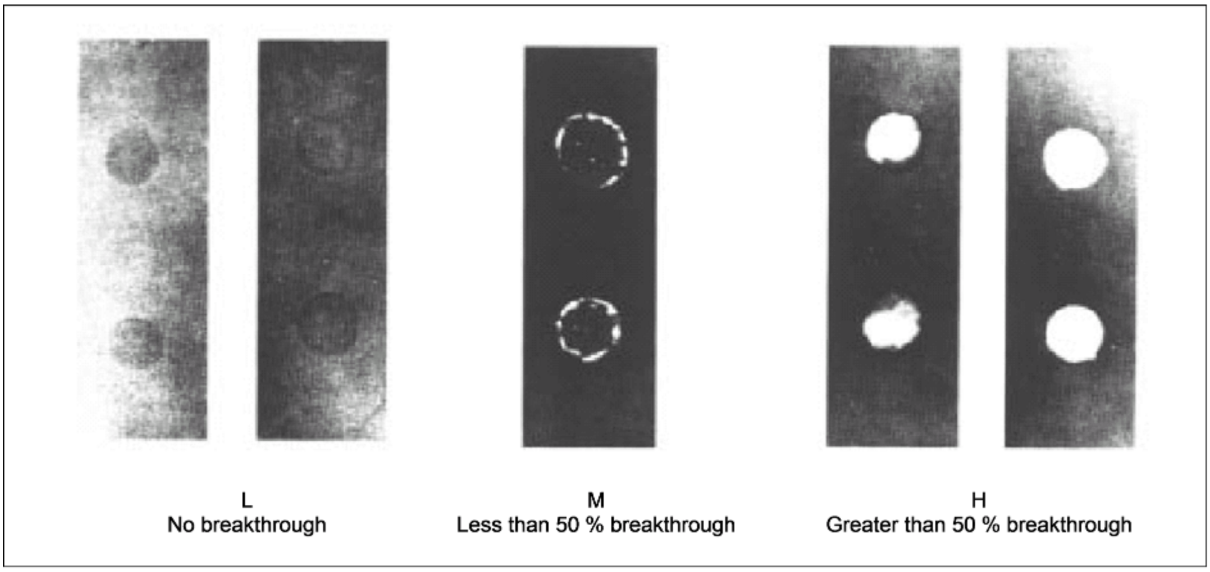


Figure 3 – Flux type classification by copper mirror test

4.10.6 Additional information

Safety: Observe all appropriate precautions on the material safety data sheet (MSDS) for chemicals involved in this test method.

4.10.7 Reference documents

ASTM E104: Maintaining Constant Relative Humidity by means of Aqueous Solutions

LLL-R-626: Rosin, Gum, Rosin Wood and Rosin Tall Oil

Sources for prepared copper mirrors:

Evaporated Metal Films, Inc., 239 Cherry Street, Ithaca, NY 14850; 607-272-3320
H. L. Clausing Co., 8038 N. Monticello Ave., Skokie, IL, 847-676-0330.

5 X: Miscellaneous test methods

5.1 Test 5-2X01: Liquid flux activity, wetting balance method

5.1.1 Object

This test prescribes the recommended test method for assessing the activity of liquid fluxes using a wetting balance.

5.1.2 Test specimen

The test specimen shall be a copper coupon complying with any acceptable industry specification. The width shall be (6,0 ± 0,25) mm wide; the length should be (25,0 ± 1) mm long or as appropriate to the test equipment. The thickness shall be (0,5 ± 0,05) mm.

5.1.3 Apparatus and reagents

The apparatus shall consist of the following:

a) a meniscus force measuring device (wetting balance) which includes a temperature-controlled solder pot containing solder maintained at (245 ± 3) °C for Sn60/Pb40 or Sn63/Pb37, or at (255 ± 3) °C for Sn96,5Ag3Cu0,5, or at 35 ± 3 °C higher than the liquidus temperature of any other solder alloy as agreed between the user and the supplier;
b) a chart recorder, data logger, or computer capable of recording force as a function of time with a minimum recorder speed of 10 mm/s;
c) a mechanical dipping device as shown in Figure 4 shall be used. This device shall be present to produce an immersion and emersion rate of (20-25) mm/s to a depth of (6,0 ± 0,1) mm and a dwell time of (5,0 ± 0,5) s.

5.1.4 Procedure

5.1.4.1 Preparation

a) The test specimen should be cleaned (degreased) by immersion in a suitable solvent, then cleaned using a (10 ± 1) % fluoroboric acid dip.
b) The coupon shall then be washed with water and dried.

5.1.4.2 Test

a) After mounting the specimen in a suitable holder, the coupon should be immersed in the liquid flux at room temperature to a minimum depth of 10 mm.
b) Excess flux is to be immediately drained off by standing the specimen vertically on a clean filter paper for 1 s to 5 s.
c) After partial drying, it should be mounted in the test equipment.
d) The surface of the molten solder shall be skimmed just prior to immersing the specimen in the solder.
e) The specimen in its holder shall be held for approximately (10 ± 1) s, 3 mm above the solder pot. The test shall be started and the specimen immersed only once using an immersion and emersion rate of (20-25) mm/s to a depth of (5,0 ± 0,1) mm and a dwell time of (5,0 ± 0,5) s.
f) During the test, the wetting curve shall be recorded on a suitable device for use in the evaluation.

5.1.5 Evaluation

Use the wetting balance curve recorded during the test to determine the following flux activity parameters:

a) A wetting time (T_w) for the wetting curve to cross the corrected zero axis after the start of the test (see Figure 5)
b) A maximum wetting force, F_{max}, taken after correction for buoyancy.

5.1.6 Additional information

5.1.6.1 General

This test method can be useful in re-qualifying materials that have exceeded the recommended shelf life. In addition, the method can help evaluate fluxing power prior to manufacturing operations on critical applications.

5.1.6.2 Safety

Observe all appropriate precautions on MSDS for chemicals involved in this test method.

5.1.6.3 Correction for buoyancy

For the wetting balance to obtain wetting force values that are relatable to one another, it is necessary to correct for the variability in specimen sizes, in particular width and thickness. This is done by correcting for the volume of the sample immersed in the solder. The following formula may be used to calculate the buoyant force correction:

$$Pb = \rho\, gV$$

where

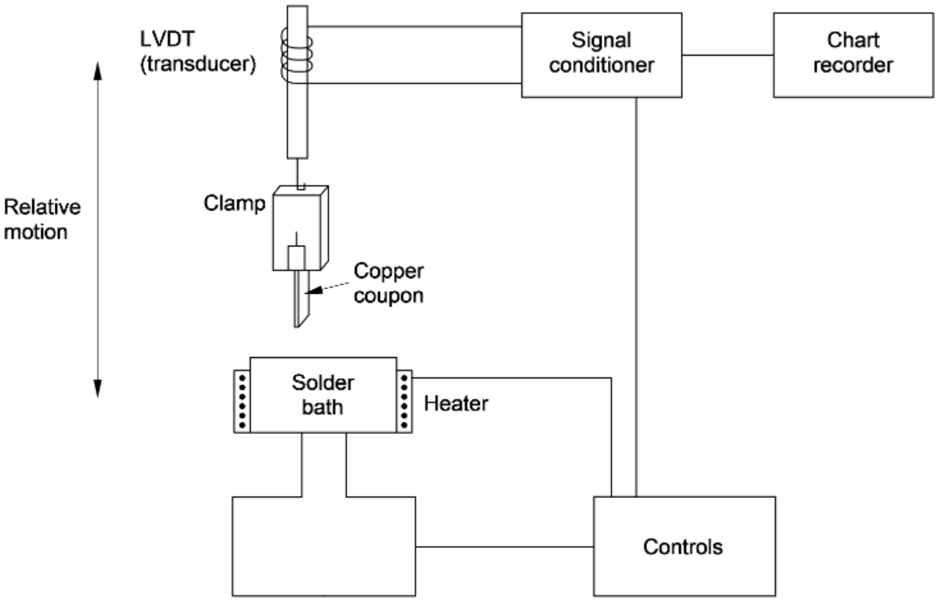
ρ is the density of solder at test temperature;

NOTE For example, 8,40 g/cm^3 for Sn63/Pb37 alloy, 7,4 g/cm^3 for Sn-3,0Ag-0,5Cu alloy.

g is the acceleration of gravity (9,81 × 10^3 mm s^{-2});

V is the immersed volume of coupon (cm^3).

When the buoyancy force is calculated it should be used to correct the zero axis. This correction is required to obtain both the proper measurement of wetting times as well as wetting forces. All measurements of wetting times and wetting forces shall be made from the corrected zero axis. In the case of an upright curve, the new corrected zero axis will be below the instrument zero (see Figures 4 and 5).



IEC

Figure 4 – Wetting balance apparatus

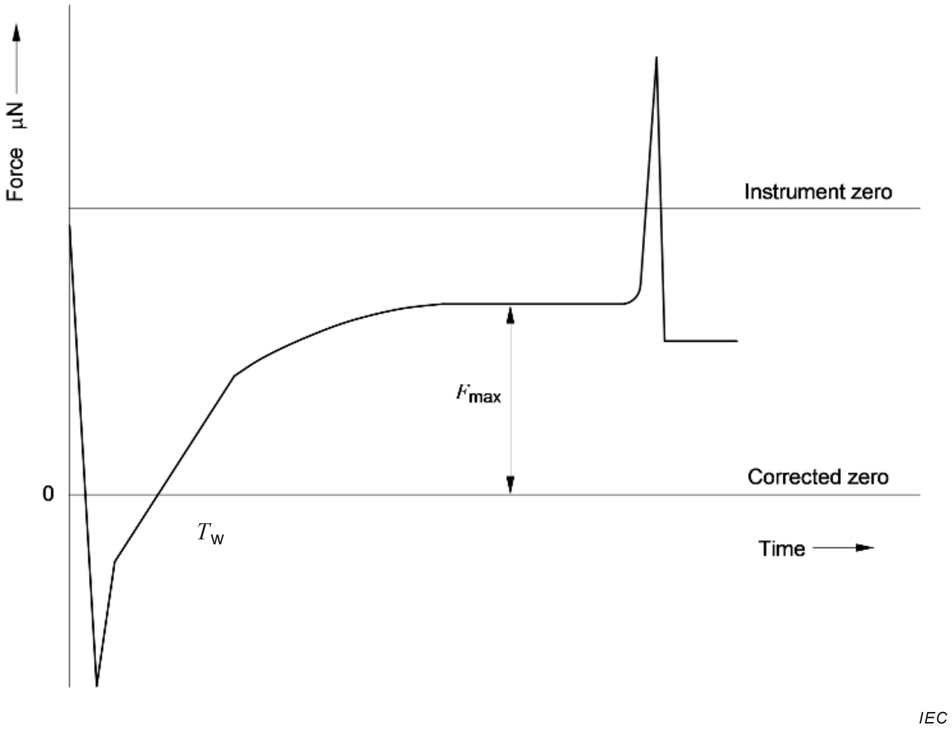


Figure 5 – Wetting balance curve

5.1.6.4 General considerations

This subclause is given for information only. The vertical force measured by the wetting balance is made up from two forces – the buoyancy force and the wetting force caused by the contact angle changing from initial non-wetting to wetting. The buoyancy force may be considered during the test, and is equal to the weight of the solder displaced, when the specimen is immersed into the solder. The only changing force is the wetting force, caused by the changing contact angle, as the specimen solders.

The corrected zero (buoyancy) line is the force when the contact angle is 90° or when the bath surface has returned to horizontal, having been initially depressed by the immersed sample. The wetting balance curve is centred on the corrected zero (buoyancy) line since the only parameter that changes during the test is the contact angle θ.

$$F = \gamma\, p \cos \theta - g\, \rho \mathrm{v}$$

Where

F is the measured force in micronewtons;

γ is the surface tension of molten solder (400 µN mm^{-1});

p is the specimen perimeter in mm;

θ is the contact angle;

g is the gravitational acceleration (9,81 × 10^{3} mm s^{-2});

ρ is the solder density (8 000 µg mm^{-3});

v is the immersed volume in mm^{3};

gpv is the buoyancy. It is the value of F when θ is 90°.

The corrected zero line (buoyancy) is a fixed reference point from which the force measurements should be taken. This line should also be used as a reference point for any time measurements. Altering the specimen dimensions changes the immersed volume and hence the buoyancy, and so alters the position of the corrected zero line; but the wetting curve still remains centred on this line. Similarly, any change in immersion depth will also alter the immersed volume, with the same effect on the buoyancy. Although use of the corrected zero line will cancel small variations in the specimen immersed volume and the immersion depth, large changes will affect the rate of heat transfer into the specimen, which will affect both T_{w}, the time to recross the corrected zero (buoyancy) line and the time to reach F_{max}.

5.2 Test 5-2X02: Spread test, liquid or extracted solder flux, solder paste and extracted cored wires or preforms

5.2.1 Object

This test method gives an indication of activity of wave solder fluxes, core solder fluxes, and solder paste. The test method offers two methods.

Method A measures the solder spread area.

Method B measures the solder spread ratio.

5.2.2 Method A

5.2.2.1 Test specimen

a) For liquid or extracted solder flux, a minimum of 10 ml that is furnished in a clean glass container.

b) For paste flux and solder paste flux, 10 ml of the diluted material (35 %).

c) For preform and cored wire, 10 ml of the extracted material.

5.2.2.2 Apparatus and reagents

a) Five replicates of 0,25 mm thick 70/30 brass of a size of approximately 40 mm × 75 mm.

b) Degreased very fine steel wool (for example, #00).

c) Solder wire from Sn63Pb37A, or Sn96,5Ag3Cu0,5, or any other solder alloy wire agreed between the user and the supplier per IEC 61190-1-3 with a diameter with 1,5 mm.

d) A solder pot not less than 25 mm in depth containing at least 2 kg solder.

5.2.2.3 Test specimen preparation

a) Clean five brass coupons with steel wool.

b) Flatten the brass coupon by bending the opposite sides of the coupon. The two bends should be parallel to the curve of the metal coil in which the brass was provided in order to stiffen and flatten the test specimen.

c) Cut a 30 mm length of solid wire solder.

d) Wrap the cut length of solder around a 3 mm mandrel.

e) Cut the coil into individual rings to make a preform of the solder.

5.2.2.4 Test

a) Maintain the solder bath at (260 ± 3) °C for Sn60Pb40, or at (255 ± 3) °C for Sn96,5Ag3Cu0,5, or at (35 ± 3) °C higher than the liquidus temperature for any other solder alloy agreed between the user and the supplier.

b) Place the preformed solder on the centre of the test specimen.

c) Place one drop (0,05 ml) of flux on the centre of the preform of the test specimen.

d) Carefully place the coupon on the surface of the solder bath for 15 s.

e) Remove the coupon in a horizontal position and place on a flat surface allowing the adhered solder to solidify undisturbed.

f) Remove all flux residue with a suitable solvent.

5.2.2.5 Evaluation

Measure the solder spread area by comparing to circles (pre-drawn) with areas similar to those listed in Table 5. The mean of the spread of all five specimens tested shall be reported.

Table 5 is intended as an aid in defining areas in mm^2.

Table 5 – Typical spread areas defined in mm^2

Diameter mm	**Area** mm^2
10,00	78,54
10,70	90,00
11,28	100,00

5.2.3 Method B

5.2.3.1 Test specimen

a) Flux may be used from several products. These may be solder paste, flux cored solder wire and liquid flux.

b) For solid flux, 25 mass % propan-2-ol or other appropriate solvent solution.

c) Solder wire of Sn63Pb37, or Sn96,5Ag3Cu0,5, or any other solder alloy agreed between the user and the supplier specified in IEC 61190-1-3 shall be wrapped on a ring bar with a diameter of 3,3 mm.

5.2.3.2 Apparatus and reagents

a) Solder bath: A solder bath with a depth of not less than 30 mm, 100 mm × 150 mm or more in width and length, provided with a temperature controller up to (50 ± 2) °C above the liquidus temperature of the tested solder.

b) Dryer: An air convection oven with a temperature controller up to (150 ± 3) °C and capable of maintaining the temperature.

c) Tongue of other proper tool suitable to lift up the test piece from the solder bath.

d) Scrubber: Suitable to remove easily the oxidized film of solder in the bath.

e) Spatula.

f) Metal mask: Thickness of 2,5 mm with a hole of 6 mm diameter.

g) Micrometer: Measurable to 0,001 mm.

h) Micro syringe or micro pipet: Measurable of 0,05 ml.

i) General experimental device: All-glass device.

j) Abrasive paper (waterproof).

k) Alcohol: Ethyl alcohol (reagent grade).

l) propan-2-ol (reagent grade).

m) Washing solvent: Proper solvent to remove the flux residue after soldering.

n) Copper plate: A plate of 50 mm × 50 mm × 0,5 mm dimensions of dephosphate copper (to prevent surface oxidation).

o) Solder: Sn63Pb37, or Sn96,5Ag3Cu0,5, or any other solder alloy agreed between the user and the supplier specified in IEC 61190-1-3 as reference specimen.

5.2.3.3 Test specimen preparation

5.2.3.3.1 Procedure of test

a) Preparation of an oxidated copper plate: The surface shall be cleaned with alcohol. One side of the plate shall be polished by abrasive paper, cleaned with alcohol, and dried thoroughly at room temperature. Put this plate into a dryer set at (150 ± 3) °C for 1 h and oxidate the plate. Four corners of the plate could be bent for easy application of a tongue.

b) Solder test specimen for liquid, solid and paste flux. The test specimen shall be one bar of 3,2 mm diameter on which wire solder of Sn63Pb37, or Sn96,5Ag3Cu0,5, or any other solder alloy wire as agreed between the user and the supplier with 1,6 mm diameter is wound.

c) Resin/rosin flux cored solder and solder paste. The product itself shall be used.

5.2.3.3.2 Preparation of test piece

a) Resin/rosin flux cored solder: After washing the face with acetone and rinsing with deionized water and then with propan-2-ol, measure and cut off (0,30 ± 0,03) g of specimen, swirl it, and place at the centre of the copper plate. Five test specimens shall be prepared.

b) Liquid flux: Measure (0,05 ± 0,005) ml from the specimen using a micro syringe or micro pipet, drop it at the centre of the copper plate, and put a solder test piece on the flux. This represents the test specimen. Five such specimens shall be prepared.

c) Paste flux: Place (0,025 ± 0,003) g of specimen at the centre of the copper plate and place the solder test piece on it. Five test specimens shall be prepared.

d) Solid flux: Adjust 25 mass % test solution with propan-2-ol or suitable solvent and measure and take (0,05 ± 0,005) ml by using micro syringe or micro pipet, and drop it at the centre of the copper plate. Place the solder piece on it. Five test specimens shall be prepared.

e) Solder paste: After stirring with a spatula the solder paste kept at room temperature, apply to the copper plate with a metal mask. Five test specimens shall be prepared.

5.2.3.4 Test

a) The test piece shall be heated while floating on a solder bath kept at (233±3)°C for Sn63Pb37, or at (255± 3)°C for Sn96,5Ag3Cu0,5, or at (35±3)°C higher than the liquidus temperature for any other solder alloy agreed between the user and the supplier, and kept at this temperature for 30 s after having fused.

b) Lift the test piece from the bath and cool it down.

c) Remove the flux residue by proper solvent.

5.2.3.5 Evaluation

The height of the spread solder fused shall be measured by a micrometer or other proper equipment. From this height, the spreading ratio shall be calculated from the formula shown below.

This procedure shall be repeated on five of the test pieces and a mean value shall be obtained, giving this as the spreading ratio of the flux representing solder under test.

$$S_{\mathrm{R}} = 100 \times (D - H)/D$$

where

S_{R} is the spreading ratio (%);

H is the height of the spread solder (mm);

D is the diameter of the solder (mm), when it is assumed to be a sphere (mm) (D = 1,24 $V^{1/3}$);

V is the mass/density of the tested solder.

Note that in the case of resin flux cored solder and solder paste, the mass of solder used for the test shall be the mass of the specimen subtracting the flux contained.

5.2.4 Additional information

Safety: Observe all appropriate precautions on MSDS for chemicals involved in this test method.

ASTM B-36 brass plate, sheet, strip, and rolled bar (according to ASTM-B-36 C2600 HO2; see Bibliography).

5.3 Test 5-2X03: Flux residues – Tackiness after drying

5.3.1 Object

This test method specifies a qualitative method for assessment of the tackiness of soft soldering flux residues. The method is applicable to fluxes of types L and M as shown in IEC 61190-1-1. The method is particularly appropriate for applications where flux residues are left in place on soldered electronic and electrical equipment.

5.3.2 Test specimen

A minimum of 0,035 g by weight is required per test for fluxes in solid or paste form. For liquid fluxes, a volume sufficient to contain a minimum of 0,035 g of non-volatile matter is required per test. For flux cored solder, a minimum 1 g is required per test and for solder paste, a minimum of 0,5 g is required per test.

5.3.3 Apparatus and reagents

a) In the test use only reagents of recognized analytical quality and only distilled, or deionized water.

b) Acid cleaning solution: Add cautiously, while stirring, 75 ml of sulphuric acid (density 1,84 g/ml) to 210 ml of water and mix. Cool, add 15 ml of nitric acid (density 1,42 g/ml) and mix the solution thoroughly.

c) Degreasing solvent, such as propan-2-ol, acetone or petroleum ether.

d) Powdered chalk.

e) 0,5 mm thick copper sheet complying with any acceptable industry specification.

f) Acetone.

g) Solder wire, or pellets, complying with IEC 61190-1-3, Sn60Pb40, or Sn96,5Ag3Cu0,5, or any other solder alloy agreed between the user and the supplier.

h) Solder bath, either circular with diameter not less than 120 mm, or rectangular with dimensions not less than 100 mm × 75 mm, containing tin-lead solder having a liquidus temperature less than 200 °C. The depth of the solder in the bath shall not be less than 40 mm. The bath shall be capable of being maintained at (235 ± 3) °C for Sn60/Pb40, or at (250 ± 3) °C for Sn96,5Ag3Cu0,5, or at (30 ± 3) °C higher than the liquidus temperature of any other solder alloy as agreed between the user and the supplier.

i) Cupping device: This shall be fitted with a 27 mm diameter die and a 20 mm diameter ball.

j) Drying oven, suitable for use at (110 ± 2) °C.

k) Tongs, or other suitable mechanical device, to lift the test piece from the surface of the molten solder bath.

l) Soft brush, of diameter approximately 7 mm.

m) Ordinary laboratory apparatus.

5.3.4 Procedure

5.3.4.1 Preparation of copper test pieces

NOTE Lettered references shown in parenthesis refer to 5.3.3.

a) From the sheet of half hard copper, approximately 0,5 mm thick (e), cut test pieces, each measuring 50 mm × 50 mm.

b) Clamp each of the test pieces, in turn, centrally onto the 27 mm die of the cupping device (i). Using the 20 mm diameter ball, make a depression in the centre of each test piece 3 mm deep, by forcing the ball into the die. One corner of the test piece may be bent up to facilitate handling with the tongs.

c) Immediately before the test, use the solvent (c) to degrease each test piece, and immerse the test pieces for 20 s in the acid cleaning solution (b). Remove the test pieces from the cleaning solution, wash well under running water, rinse in acetone (f) and dry by air blowing at room temperature.

d) Test for solid, paste and liquid flux samples

   - Weigh (1,00 ± 0,05) g of the solder wire or pellets (g), previously degreased in the solvent (c), and transfer it to the centre of the depression in one of the cleaned copper test pieces. This may conveniently be done, if solder wire is used, by forming the wire into a tight spiral.
   - If the flux under test is in solid or paste form, weigh between 0,035 g and 0,040 g of the solid or paste flux and add this to the solder in the depression of the test piece.
   - If the flux under test is in liquid form, first determine its non-volatile matter content by the use of the method described in ISO 9455-1 and ISO 9455-2. Then add the appropriate volume of the liquid flux, containing between 0,035 g and 0,040 g of non-volatile matter, to the solder in the depression of the test piece. Evaporate the solvent at 60 °C for 10 min in the drying oven. If the liquid flux has low non-volatile content, it may be necessary to add the flux in two increments, carrying out the evaporation procedure after each addition.
   - For flux cored solder samples: Degrease the surface of a suitable length of the cored solder sample, using a cloth dampened with the solvent (c). Weigh (1,00 ± 0,05) g of the degreased sample, form it into a small flat coil and place it in the centre of the depression in one of the cleaned copper test pieces.
   - For solder paste samples: Weigh (0,50 ± 0,05) g of the solder paste sample into the centre of the depression in one of the cleaned copper test pieces.

5.3.4.2 Heating the test piece

NOTE Lettered references shown in parenthesis refer to 5.3.3.

a) Using the tongs (k), or other suitable means, carefully lower the prepared test piece onto the surface of the molten solder, maintained at the test temperature in the solder bath (h). Test temperature shall be at (235 ± 3) °C for Sn60/Pb40, or at (250 ± 3) °C for Sn96,5Ag3Cu0,5, or at (30 ± 3) °C higher than the liquidus temperature of any other solder alloy as agreed between the user and the supplier.

b) Allow the test piece to float on the solder bath until the solder melts and leave the test piece in this position for a further 5 s. Remove the test piece carefully from the bath and allow it to cool, in air, in a horizontal position for 30 min.

5.3.4.3 Examination of the test piece

Dust the surface of the flux residue on the test piece liberally with the powdered chalk (d). Lightly brush the chalked surface with the soft brush (l).

5.3.5 Evaluation

If the chalk powder is easily removed by brushing, the flux is deemed to be "not tacky." If the chalk powder cannot be removed by brushing, or can be removed only with difficulty, the flux is deemed to be "tacky."

5.3.6 Additional information

Safety: Observe all appropriate precautions on MSDS for chemicals involved in this test method.

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IEC 60068-2-20, *Environmental testing – Part 2-20: Tests – Test T: Test methods for solderability and resistance to soldering heat of devices with leads*

IEC 61189-1, *Test methods for electrical materials, interconnection structures and assemblies – Part 1: General test methods and methodology*

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IEC 61189-3, *Test methods for electrical materials, printed boards and other interconnection structures and assemblies – Part 3: Test methods for interconnection structures (printed boards)*

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ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results – Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

ISO 9001*, Quality management systems – Requirements*

IPC-9201:1996, Surface Insulation Resistance Handbook

IPC-TR-467:1996, Supporting Data & Numerical Examples for J-STD-001B (Control of Fluxes)

ASTM B-36 Brass Plate, Sheet, Strip, and Rolled Bar

ASTM E104: Maintaining Constant Relative Humidity by means of Aqueous Solutions

LLL-R-626: Rosin, Gum, Rosin Wood and Rosin Tall Oil

Evaporated Metal Films, Inc., 239 Cherry Street, Ithaca, NY 14850; 607-272-3320

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